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(71) Applicant (*for all designated States except US*): **ABB BOMEM INC.** [CA/CA]; 585, boul. Charest Est, Bureau 300, Québec, Québec G1K 9H4 (CA).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **BORDELEAU, Alain** [CA/CA]; 1148, Robert L. Séguin, Sainte-Foy, Québec G1X 4P5 (CA). **BUIJS, Henry** [CA/CA]; 2051, Dickson, Sillery, Québec G1T 1C6 (CA). **ROBERGE, Raymond** [CA/CA]; 611, rue Calais, St-Nicolas, Québec G7A 1L6 (CA). **SAMSON, Perry** [CA/CA]; 1316, Esplanade, Val-Bélair, Québec G3J 1E8 (CA).

(74) Agents: **MARCOUX, Paul** et al.; Ogilvy Renault, Suite 1600, 1981 McGill College Avenue, Montreal, Québec H3A 2Y3 (CA).

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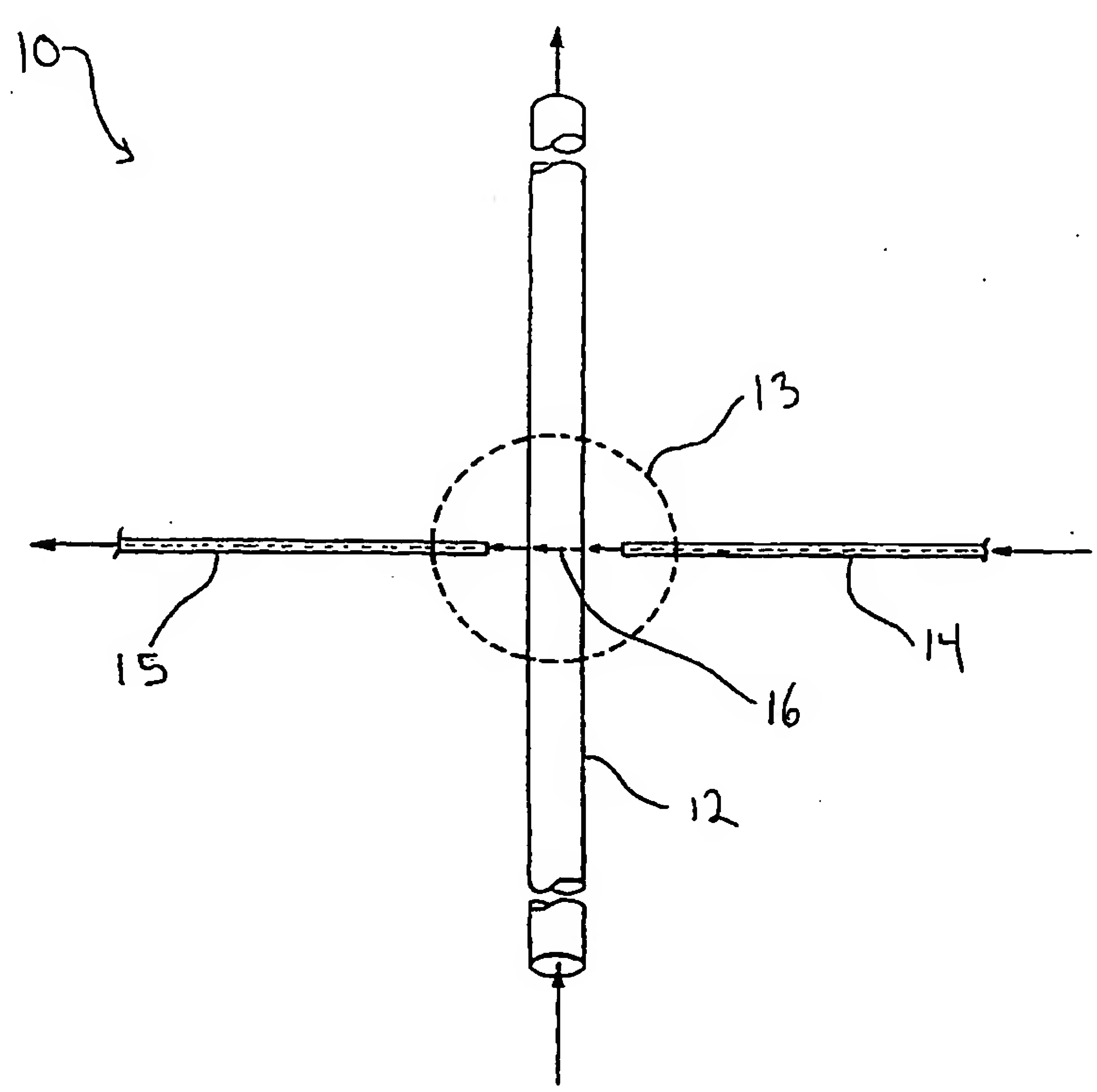
(54) Title: FLOW-THROUGH CELL



(57) Abstract: The present invention relates to flow-through cell for qualitative or quantitative optical analysis of physical or chemical properties of different liquid, fluid, paste, or powder compositions, using a conveying conduit essentially composed of PTFE or other optically transparent or translucent material, such as other fluorinated polymers, and through which passes a light pathway. The light pathway can passes through an entire conveying conduit or through a section thereof. The light wavelength used in this flow cell apparatus can be selected from infrared to visible light spectra. The light passing through the composition is received, transmitted and translated into an interpretable form.

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FLOW-THROUGH CELL

BACKGROUND OF THE INVENTION

(a) Field of the Invention

The invention relates to a flow-through cell to be used in optical spectroscopic analyzers. Most particularly, it relates to instruments and methods for analyzing the chemical compositions and physical properties of any material that can circulate in a tube-like conveying conduit composed principally of visible light and infrared transparent optical material such as polytetrafluoroethylene (PTFE) or other transparent fluorinated hydrocarbon polymers. The invention allows for measuring and analyzing the visible and infrared light absorption at various wavelengths to determine the chemical composition and physical properties of the tested material.

(b) Description of Prior Art

Infrared Spectroscopy

A common way to analyze materials is by spectroscopy using electromagnetic radiation. A beam of radiation consisting of various wavelengths is passed through a well-defined thickness of a material and the loss of intensity as a function of the wavelengths is determined.

In the wavelength range where unique absorption patterns occur (also called the "mid infrared (IR)" or "fingerprint" region), the absorptivity is usually quite high. In order to avoid absorbing all the incident radiation in the sample, the optical path through the sample must be short. It physically ranges from less than 10 micrometers for some aqueous solutions to 0.1 mm for weakly absorbing organic species or diluted samples.

Many sampling devices have been developed for assisting the laboratory technician measuring a sample with an infrared spectrometer. They are principally designed for manual loading of the sample. As a result, these sampling devices are unsuitable for automated infrared

analysis. Two exceptions are a flow through cell for liquid sampling and a diffuse reflectance accessory for the analysis of solids in powder or grain size. The liquid cell consists of a body through which liquid can flow past a pair of infrared transparent windows from an inlet to an outlet. The windows are placed such that the path length of the radiation through the product is fixed at a length best suited for the application.

For mid infrared analysis, the space between the inner faces of the windows currently used in the industry is set typically at from 10 to 100 micrometers and needs to be well controlled. This limits the amount of sample that can flow past the IR beam.

Mid infrared vs. Near infrared (NIR)

The observed infrared absorption bands are manifestations of the changes in the fundamental vibrations of a molecule induced by the incident IR radiation. Molecular vibrations have harmonic distortion; this will create transitions at multiples and combinations of the fundamental frequencies as well. These multiples are harmonics or harmonic frequencies and combinations of the fundamental vibrations. The absorptivity at the harmonic and combination frequencies is much lower than the fundamental frequencies. This permits the use of an increased path length of the IR beam through the sample.

Infrared analysis at the harmonic overtones of the fundamental frequencies and combination frequencies is called Near IR spectroscopy. It is commonly used for automated analysis in factories in the chemical, pharmaceutical and food processing industries. The Near IR beam can traverse a substantial distance through a sample permitting the sampling of more product. At the longer wavelength end of the Near IR, the path length of optimum transmission is up to 1 mm while at the short wavelength end it can be more than 1 cm.

Flow through cells

A common practice for constructing flow through cells is to make the cell body from a strong inert material. Stainless steel is frequently

used. This cell body is then provided with pipe couplings at the inlet and outlet to connect the cell to a fluid circuit. IR transparent windows are placed in the cell body with a gasket seal such that the IR beam traverses the sample perpendicular to the flow direction.

A gasket seal between the IR transparent windows and the stainless steel body often causes problems of loading fluids out of the cell. Different reasons can explain these problems: 1) the gasket seal degrades due to chemical incompatibility and high temperature of the product being analyzed and the materials used causing the seal to leak: 2) differential expansion-contraction between the cell body and the windows can cause the seal to fail. The coefficients of thermal expansion of the cell body material and the windows are usually different. Another disadvantage of a gasket seal is that crevices created by the seal will retain sample and cause contamination or difficulty with cleaning.

KBr and NaCl are salt crystals that have high mid IR transmission. These materials are therefore a choice for flow through cell bodies, which transmit IR. However these materials are water-soluble and therefore cannot be used with aqueous solutions or in high humidity environments. Also their mechanical properties are not amenable to being machined in complex shapes.

U.S. Pat. No. 4,575,424 discloses flow cells for light absorption detectors that are internally coated with rhodium, which is polished to a mirror finish. However, the flow cell coating is a rather expensive material which requires complicated manufacturing techniques to give a highly polished surface on the inside of a narrow bore. Further, rhodium is not a perfect electrical conductor, and, consequently, there is some absorption of light upon reflection.

U.S. Pat. No. 6,069,094 discloses flow cells composed of a fluid passageway, opposed optic cable assemblies with sapphire windows. The optic fiber cable assemblies are housed in stainless steel tubes that project into the flow cell and that have distal ends sapphire windows. By means of

feeler gauge fixture and feeler gauges, the gap between the distal ends of the optic fiber cable assemblies can be set to a desired path length for the infrared radiation to travel through the liquid sample.

There has been another method that utilizes a flow cell having a flow passage of a specific form with a measuring portion provided therein to classify and analyze particles in a liquid. This method, in the flow passage of the flow cell, forms a flow of the liquid to be measured, which is surrounded by a sheath flow, and measures particles in the sample liquid by an optical analyzer. Such a method is suitable to automate the examination of particles in a liquid. For example, an apparatus in which a fluid sample is introduced into a flow passage of a specific shape and particles in the sample are photographed in a wide imaging region is known. Through analysis of the still images thus obtained, the particles in a liquid can morphologically be analyzed. Further, the concentration of the particles may be analyzed by counting how many particles are contained in a volume of the photographed sample.

U.S. Pat. No. 5,360,004 (Purdy et al.) describes a method and apparatus for the determination of blood analyte concentrations, wherein a body portion is irradiated with radiation containing two or more distinct bands of continuous-wavelength incident radiation. Purdy et al. emphasize filtration techniques to specifically block radiation at the two peaks in the Near IR absorption spectrum of water, occurring at about 1440 and 1935 nm. Such selective blocking is carried out in order to avoid a heating effect that may be due to the absorption of radiation by water in the body part being irradiated.

U.S. Pat. No. 5,267,152 (Yang et al.) describes noninvasive devices and techniques for measuring blood glucose concentration using only the portion of the IR spectrum that contains the Near IR water absorption peaks (e.g., the "water transmission window," which includes those wavelengths between 1300 and 1900 nm). Light is directed to a tissue source and then collected by an integrating sphere. The collected

light is analyzed and blood glucose concentration is calculated using a stored reference calibration curve.

U.S. Pat. No. 5,242,602 (Richardson et al.) describes a method for analyzing aqueous systems to detect multiple active or inactive water treating components. The method involves determination of the absorbency or emission spectrum of components over a range of 200 to 2500 nm, and the application of chemometrics algorithms to extract segments of the spectral data obtained to quantify multiple performance indicators.

U.S. Pat. No. 5,252,829 (Nygaard et al.) describes a method and apparatus for measuring the concentration of urea in a milk sample using an infrared attenuation measuring technique. Multivariate techniques are carried out to determine spectral contributions of known components using partial least square algorithms, principal component regression, multiple linear regression or artificial neural network learning. Calibration is carried out by accounting for the component contributions that block the analyte signal of interest. Thus, Nygaard et al. describes a technique for measuring multiple analyte infrared attenuations and compensating for the influence of background analytes to obtain a more accurate measurement.

U.S. Pat. No. 4,975,581 (Robinson et al.) describes a method and apparatus for determining analyte concentration in a biological sample based on a comparison of infrared energy absorption (i.e., differences in absorption at several wavelengths) between a known analyte concentration and a sample. The comparison is performed using partial least square analysis or other multivariate techniques.

U.S. Pat. No. 4,882,492 (Schlager) describes a method and apparatus for non-invasive determination of blood analyte concentrations. Modulated IR radiation is directed against a tissue sample (e.g., an ear lobe) and either passed through the tissue or impinged on a skin surface where it is spectrally modified by a target analyte (glucose). The spectrally modified radiation is then split, wherein one portion is directed through a

negative correlation cell and another through a reference cell. Intensity of the radiation passing through the cells are compared to determine analyte concentration in the sample.

U.S. Pat. No. 4,306,152 (Ross et al.) describes an optical fluid analyzer designed to minimize the effect of background absorption (i.e., the overall or base level optical absorption of a fluid sample) on the accuracy of measurement in a turbid sample or in a liquid sample which is otherwise difficult to analyze. The apparatus measures an optical signal at the characteristic optical absorption of a sample component of interest and another signal at a wavelength selected to approximate background absorption, and then subtracts to reduce the background component of the analyte-dependent signal.

Considering the state of the art mentioned above, there is still needs of apparatus allowing the analysis of different material passing into a flow through cell. It would be highly desirable to be provided with a device and a method for monitoring or controlling physical and chemical properties of different kinds of manufacturing processes, which requires data on the concentration, composition, size distribution or shape of particles on a quick and continuous basis. Therefore, it is clear that the best way of obtaining the data would be to use accurate highly sensitive and versatile optical sensor, which would allow an analyzer setting to measure directly from a process conveying conduit, pipe or container. The measured sample would give reliable information on the process.

SUMMARY OF THE INVENTION

One object of the present invention is to provide a flow-through cell for optical analysis of a composition comprising:

- at least one monolithic conveying conduit for transporting the composition;
- at least one input light source for illuminating the composition through a section of the conveying conduit; and

- a light receiving sensor, the light receiving sensor collects light emerging through the illuminated composition and capable of transmitting received light transmitted through a composition to an analyzer operatively connected to produce an output indicative of the quantitative and/or qualitative level of light transmitted through the composition.

The input light source may be perpendicular to the conveying conduit.

Another object of the present invention is to provide A method for measuring quantity or concentration of at least one component or physical property of a composition comprising:

a) capturing light emerging through an illuminated composition passing through a monolithic conveying conduit: and

b) comparing said captured light of step a) with said light before its passage through said composition to obtain a differential measure of light indicative of the quantity or concentration of a component or physical property of said composition.

Another object of the present invention is to provide a flow-through cell wherein the monolithic conveying conduit is made with matter selected from the group consisting of polytetrafluoroethylene, PTFE, borosilicate aluminosodique, glass, fluorinated hydrocarbons, quartz, silica, sapphire, and transparent matter allowing passage of visible light, infrared and ultra violet radiation.

The light that may be used in the present invention may be visible light, infrared or ultra-violet radiation.

The infrared may be composed of wavelength in the range of 600 nm to 25000 nm.

In accordance with the present invention there is provided a flow-through cell permitting the analysis of physical and/or chemical

properties of a composition that may be selected from the group consisting of liquid, gas, air, fluid, paste, foam, emulsion, gel, and powder.

The flow-through cell according to the present invention may further comprise means for holding the conveying conduit in a desired position relatively to a light pathway traversing the conveying conduit.

Another object of the present invention is to provide a flow-through cell wherein the means for holding the conveying conduit can be variably positioned to adjustably reduce the optical path through the conveying conduit.

The flow-through cell according to the present invention may further comprise a mean for modulating the temperature of the composition before and/or during passage through a measuring section.

For the purpose of the present invention the following terms are defined below.

The term "conduit" as used herein is intended to mean a tube, a pipe, a container, a channel, a tunnel, or a duct that is used for conveying any composition to be analyzed.

The term "receiving sensor" as used herein is intended to encompass any optical electronic surface that converts visible light, UV, or IR radiation transmitted through a sample of material under test to an intensity distribution as a function of wavelength. The intensity distribution may be correlated with selected chemical composition or physical properties either by intensity variations at specific wavelengths or by a partial least square analysis or principal component regression.

The term "monolithic conveying conduit" is intended to mean a conduit cast as a single piece, formed from a single matter or composed of material without joints, windows, seams, or seals. A monolithic conveying conduit may include any matter or composition allowing passage of visible light, infrared or ultra violet radiation in the analysis section, or in the section where passes the light or radiation through the material to be

analyzed. The monolithic conveying conduit may result from milling, grinding, machining or forming optically transparent or translucent material.

The expression "physical properties" may be intended to mean herein properties including viscosity, consistency, density, or concentration.

For the purpose of the present invention, a receiving sensor generally has absorption characteristics that are derived from a partial least square analysis or principal component regression analysis. The receiving sensor may be used to selectively emphasize wavelengths having high correlation with a selected physical characteristic or component concentration in a composition. For example, but not limited to, the quantitative association between the absorption spectrum at a particular wavelength and a particular component concentration does a correlation.

The term " analyzer" as used herein is intended to mean a device or apparatus that can translate the quantitative and/or qualitative level of light transmitted through an illuminated composition.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 illustrates a schematic perspective view of the flow-through cell in accordance with an embodiment of the present invention,

Fig. 2 illustrates a schematic perspective view of the flow-through cell in accordance with another embodiment of the present invention;

Fig. 3 is a section view of the conveying conduit in accordance with an embodiment of the present invention;

Fig. 4 illustrates according to another embodiment of the present invention a transversal section view where passageways are inwardly extending into the lumen of the conveying conduit;

Fig. 5 illustrates Distribution of light intensity verses wavelength through Monolithic conveying conduit made from PTFE filled with flowing milk. The spectral region covers the near infrared from 5400 to 8000 cm-1 (1852nm to 1250nm);

Fig. 6 illustrates Demonstration of analysis of fat and protein in milk flowing through a Monolithic-conveying conduit made from PTFE material;

Fig. 7 illustrates Determination of % moisture in butter by Near Infrared analysis using a monolithic conveying conduit machined from a single block of PTFE material. The graph shows repeated determinations at intervals of 1 minute. The square, round and triangular dots indicate independent duplicate and average laboratory determinations of moisture for samples extracted from the process at the time indicated;

Fig. 8 illustrates Photograph of monolithic cell. The cell machined from PTFE is enclosed in a stainless steel housing for connection to other stainless steel piping and for protection. In this example the process pipe has a diameter of 4 inch (10cm). Other sizes can be employed to match the size and flow of the process pipe to which it is connected;

Fig. 9 illustrates Distribution of light intensity verses wavelength through Monolithic conveying conduit made from PTFE filled with a variety of meat products including lean and fat Pork and lean and fat beef. The spectral region covers the near infrared from 7000 to 16000 cm-1 (1428nm to 625nm); and

Fig. 10 illustrates Photograph of an installation of monolithic conveying conduit machined from a block of PTFE infrared transparent material for the analysis of flowing ground meat. The cable attached to the middle of the cell is a fiber optic cable, which transports infrared light that has been modulated with multiple unique frequencies corresponding to the wavelengths of the light as produced by an FT-NIR spectrometer. The light

is measured by an infrared detector on the opposite side of the cell and decoded by means of a Fourier Transformation performed in a computer in order to produce a spectrum as shown in figure 1-ex3. The infrared spectra contain variations that vary linearly with the concentrations of different quality parameters such as % fat, % protein and % moisture. The concentrations are determined by a Partial Least Squares multivariate analysis.

DETAILED DESCRIPTION OF THE INVENTION

In accordance with the present invention, there is provided an apparatus and method for optically analyzing the chemical composition and physical properties of different materials.

Turning to Fig. 1, according to the illustrated embodiment, the flow cell 10 comprises a conveying conduit 12 which allows passage of a composition to be analyzed through a measuring section 13, a light source 14, and a light receiving sensor 15. The conveying conduit 12 is essentially composed of optically transparent or translucent material such as polytetrafluoroethylene (PTFE), other fully fluorinated hydrocarbon polymers, aluminosodic borosilicate, and any other light and IR transmitting material. Typically, the conveying conduit 12 will be cylindrical. The composition to be analyzed passes into the conveying conduit 12, through a light pathway 16 extending between the light source 14 and the light-receiving sensor 15. The light that is transmitted by the material to be analyzed is received at the receiving sensor 15. One of ordinary skill in the art will recognize that the flow cell 10 can take various other shapes and geometries besides that depicted in Fig. 1.

Fig. 2 shows another embodiment of the flow cell 10 that comprises a conveying conduit 12 held on a casing 18 by securing flanges 20 and 22, for conveying a material to be analyzed through a light pathway 16, a light source 14, and a light receiving sensor 15. The light source 14 and the light-receiving sensor 15 are secured to the casing 18 with the flanges 24 and 26. The conveying conduit 12 may be squeezably held

and/or tightened by jaws 28. The distance between jaws 28, in the measuring section 13 in which passes the light pathway 16, may be adjusted with a screw like crank 30. Securing hooks 42 may be used to fix a cover placed the casing 18 to keep the inside of the flow cell 10 protected from external light that can interfere with the light pathway 16.

As shown in Fig. 3, according to another embodiment, a conveying conduit 12 comprises a portion 32 having a shorter distance than the remaining portion of the conveying conduit 12. The conveying conduit 12 comprises partial passageways 38 and 40 in which are partially inserted light source 14 and receiving sensor 15 in a manner to optimize the passage of the light pathway 16 through the composition 36 passing in the portion 32.

According to another embodiment of the present invention as shown in Fig. 4, a conveying conduit 12 comprises inwardly extending passageways 42 and 44 into the lumen 46 of the conveying conduit 12. The inwardly extending passageways 42 and 44 allow passage of a light pathway 16 by a light emitting source 14 through composition 36 and capture of light portion passed throughout composition 36 by a receiving sensor 15. It will be comprised by those skilled in the art that inwardly extending passageways 42 and 44 may be configured for having different sizes, diameters, forms and lengths. Also, the distance between extremities 48 of inwardly extending passageways 42 and 44 can be settled depending of the needs.

In another embodiment, the present invention provides a flow cell for determining physical and chemical properties, such as but not limited to, viscosity, consistency, composition, or concentration. The present invention may use all spectral information contained in the visible light or in the near-IR region in order to obtain a set of measurements that can be used to determine the characteristics of a composition.

Another embodiment of the invention consists in providing a flow through cell made of visible light and IR transparent material such that no windows or window seals are needed.

One embodiment is to provide a PTFE or other fluorinated polymer material flow through cell for optical analysis. Clear PTFE, without color producing fillers, has excellent transmission at all wavelengths in the visible light and near IR region of the spectrum.

A particular range of wavelengths according to the present invention is the infrared where many substances have characteristic patterns of absorption as a function of wavelength. For quantitative determination of different chemical components making up a substance, the Beer-Lambert law provides a linear relationship between concentration and absorption intensity. The constant of the equation is the product of path length the radiation traverses through the product and the absorptivity of the product.

According to the present invention, another embodiment consists in confining a length of flexible PTFE tubing between two jaws to form two approximately flat faces at the point in the center of the jaws where the Near IR beam is directed through the walls of the tubing and the flowing material to be analyzed. In this way, an optical flow through cell is provided where no separate window or window seals are needed. The jaws are shaped such as to gradually squeeze the PTFE tubing in one direction. The spacing between the jaws can be adjusted to achieve the path length best suited for the application. For example, but not limited to, a nominal 10 mm inner diameter (ID) PTFE tubing can be squeezed down to form a cell with spacing of from 8 to less than 1 mm IR path length. Connections to a fluid system can be done directly with the PTFE tubing cut to the required length or via other piping coupled to the PTFE tubing of the cell.

An advantage of the cell constructed from PTFE is that it is chemically inert and is low wetting; an inherent property of PTFE. Further

there are no seal crevasses where sample can be retained or where flow can be hindered. This permits qualifying the cell for analysis of food products such as milk where in-place cleaning (CIP) is desirable. There is no possibility of leakage as long as the pressure and temperature specifications of the tubing are respected.

The PTFE conveying conduit, which may be in the form of a portion of a tube, a groove, a square chamber or any other form, may have dimension between 0.1 mm to about 20 cm at the level of the light pathway.

In a particular embodiment of the present invention, the conveying conduit is in the form of a PTFE tube, rated for pressures typically up to 10 Bar and temperatures typically up to 150°C.

Because of the low wetting property of PTFE, the PTFE tube cell is suitable not only for the analysis of pure liquids but also liquids with solid suspensions such as milk and pasty materials such as butter and process cheese that have a tendency to stick to many types of material.

In another embodiment using PTFE as the cell material, the cell is machined from a block of solid PTFE. The advantages are the same as for the above PTFE tube cell but in addition there is more freedom in the form it can have. Machining the cell from a solid block of PTFE permits machining standard pipe coupling ends (such as triclover and others) at the inlet and outlet. Also machining the cell from a PTFE block permits making cells for larger pipes than is possible with PTFE tubing.

The flow through cell according to the present invention may comprise a monolithic conveying conduit made of different transparent or translucent matter, such as, but not limited to fluorinated hydrocarbons, quartz, silica, sapphire, aluminosodic borosilicate, glass, and transparent matter allowing passage of visible light, infrared and ultra violet radiations.

In one embodiment of the present invention, there is provided a light receiving sensor capable of collecting light emerging through an illuminated composition and capable of transmitting received light

transmitted through the composition to a sensitive analyzer operatively positioned to produce an output indicative of the quantitative and/or qualitative level of light transmitted through the composition. The analyzer may be any setup, or apparatus able to convert the transmitted light message from the light-receiving sensor in data that can be interpreted.

Still another embodiment of the invention is to provide a flow cell that can change the width of a sample flow.

Still another embodiment of the invention is to provide a flow cell that can change the shape of a sample flow.

Still another embodiment of the invention is to provide a flow cell, wherein the essential portions prevent waste or particle accumulation on the inner wall surface or, if needed, can easily be cleaned in a short time and the maintenance of which is easy to be made.

Still another embodiment of the invention is to provide a flow cell which can allow control of the width, the thickness and the velocity of a sample flow to cause the sample flow to pass within a measuring range and in order to make the flow velocity at the testing portion and that at the peripheral portion to be uniform.

The invention is also intended to form, in a measuring portion, or analysis section, a sample flow of a fixed width which is less in turbulence and is free of unevenness in flow velocity between the central portion and the peripheral portion. If the flow is stable even at high velocity, the sample liquid can be properly measured.

According to the invention, a flow through cell provided for measuring particles suspended in a liquid, comprises a light or IR translucent or transmitting conveying conduit supplying a sample composition to be measured. The flow cell has a transparent conveying conduit without any windows for providing an unobstructed view of the flow passage in at least one direction which is transversal of the flow cell.

In another embodiment of the present invention, more than one light pathway can be comprised in the flow cell. The light sources that may

produce light of different wavelengths may be placed side by side in perpendicular position along a conveying conduit, therefore allowing analysis of several at the same time or analysis of different parameters of a component under different wavelengths.

With small bore PTFE tubing, an automated optical analysis may be mounted parallel to a side stream of a larger process stream.

In one embodiment, a monolithic flow through cell can be made large enough to directly fit in a main process pipe. The IR beam may give measures corresponding to products passing through a process pipe, as the conveying conduit may have a dimension of up to about 20 cm at the level of the light pathway. The passage through the cell is shaped such that the cross section includes a passage with 10 mm or less space between walls. This is the area with light pathway for IR analysis. At the same time the passage through the cell is gradually reduced by a predetermined percentage of the inlet and outlet cross section. This is to force a maximum amount of material through the gap where the IR analysis is done without excessively impeding flow. The ratio of cross section where the IR beam traverses to total cross section determines the fraction of product analyzed by IR.

The present invention thus provides an analyzing device by means of which an accurate quantitative and qualitative analysis of the chemical composition can be carried out directly from a main flow or adapted passageway, as for example but not limited to, a groove machined into the conveying conduit's wall. The illuminated volume portion passing into this groove is accurately defined and easily adjustable.

In another embodiment, the flow through cell may be provided with heating plates inserted in grooves machined in the PTFE block cell to control sample temperature in the cell or to raise the temperature in the cell to reduce viscosity of high viscosity products as they pass through the cell.

In its most simple configuration, the flow-through cell of the present invention, as shown in Fig.1, is constituted of a light emitting source emitting light in a path through a PTFE or fluorinated conveying conduit, and a light receiving sensor receiving the light having passed through a transversal tube. All these part of the flow-through cell being fixed or secured simply on a surface and used in a dark environment.

In another configuration of the flow-through cell, the inlet outlet connection areas may be surrounded by metal sleeves with thread or other connections, such as Tri-Clover™, to be compatible with standard food process pipes.

Additionally, there may be provided an opaque cover surrounding the PTFE block cell to avoid stray ambient light from interfering with the IR beam for analysis.

Another embodiment is the possibility to bring the visible or IR beam from a remote spectrometer to the cell and returning the transmitted beam back to the spectrometer via fiber optic cables. It is also possible to bring the visible or IR beam with wavelength selection information from a remote spectrometer to the cell and analyzing the spectrum with a light or radiation detector placed directly after the cell. This increases the measured light or radiation intensity because the beam does not need to be refocused on a small optical fiber end. This is advantageous for the optical analysis of scattering samples such as for example but not limited to, milk, food products, laboratory products, or industrial fluids, where the beam after transmission through the cell is scattered to a large diameter. This is called analysis by "diffuse transmission".

For the application of diffuse transmission analysis of scattering samples, all the above embodiments can be realized with PTFE material containing scattering filler such as, but not limited to, titanium dioxide. This has the advantage that no signal saturation will occur when the scattering sample is not present. This also prevents a common problem of detector

blindness when sample flow is discontinuous such as for ground meat analysis or dry cracked cheese.

In some analysis situations sticking or stagnation of sample may still occur despite the non-wetting property of PTFE. An added implementation is an air jet just upstream from the gap where the light or radiation beam passes through the PTFE walls and the sample material. Introduction of air at regular intervals at a short duty cycle is effective for the removal of stagnant sample.

It may be also desirable to perform automated optical analysis on flowing material that is at high temperature. For example, but not limited to, the production of polyester polymer material in liquid form is done at a temperature of approximately 275°C. The present invention may make use of a flow through cell out of low thermal expansion glass such as Pyrex™, or borosilicate aluminosodique glass. The glass like PTFE is transparent at all Near IR wavelengths.

The construction principle is the same as for the PTFE tube cell shown in Fig. 2 except that the cell body is rigid glass and does not need supporting jaws. Any reduction of the optical path from the nominal inside diameter of a piece of Pyrex™ tubing is achieved by deforming the tubing at high temperature instead of squeezing between jaws. Pyrex™ remains rigid at temperatures well above 300°C. So no shape support is needed.

In one embodiment of the present invention, the flow rate of a composition, temperature and pressure applied thereon in the conveying conduit may be variably controlled depending of needs.

In another embodiment of the present invention, the flow-through cell is installed on a production line and utilized to provide data for quality control.

The present invention will be more readily understood by referring to the following examples, which are given to illustrate the invention rather than to limit its scope.

EXAMPLE I

Milk analysis

Near Infrared Analysis of flowing milk and milk products through a monolithic conveying conduit made from PTFE material.

Analysis of the percentage of milk fat and percentage protein in normal dairy milk has been demonstrated using Near Infrared spectroscopy.

Fig. 5 shows the distribution of light as a function of wavelength through a sample of flowing milk through a Monolithic conveying conduit made from a section of tubing with 10mm inside diameter manufactured from PTFE material. Distribution of light intensity verses wavelength through Monolithic conveying conduit made from PTFE filled with flowing milk. The spectral region covers the near infrared from 5400 to 8000 cm-1 (1852nm to 1250nm).

Fig. 6 shows the results of analysis demonstrating the determination of percentage fat and percentage protein for different small quantities of milk flowing through the tubing. Demonstration of analysis of fat and protein in milk flowing through a Monolithic-conveying conduit made from PTFE material.

Precise determination of percent fat and percent protein in dairy milk has been demonstrated with the use of a Monolithic conveying conduit connected to a Near infrared spectrum analyzer and detection system.

The advantage of the flow cell as described in the invention is that the conduit is made from material that is easy to clean and maintain in sanitary condition for the purpose of handling milk for human consumption. There are no crevasses or stagnant areas where milk product can accumulate and form bacteria. The cell is designed such that it meets accepted standards of sanitation established for food handling equipment

with cleaning procedures that do not require dismantling of the assembly: Certified for clean in place CIP.

EXAMPLE II

Butter analysis

Continuous analysis of butter during manufacturing as the butter flows through a large bore monolithic conveying conduit machined from a single block of PTFE material.

Fig.7 Determination of % moisture in butter by Near Infrared analysis using a monolithic conveying conduit machined from a single block of PTFE material. The graph shows repeated determinations at intervals of 1 minute. The square, round and triangular dots indicate independent duplicate and average laboratory determinations of moisture for samples extracted from the process at the time indicated.

Fig. 8 shows a photograph of monolithic cell. The cell machined from PTFE is enclosed in a stainless steel housing for connection to other stainless steel piping and for protection. In this example the process pipe has a diameter of 4 inch (10cm). Other sizes can be employed to match the size and flow of the process pipe to which it is connected.

The advantages of the cell design as described in this invention is that it permits determination of quality parameters for butter on a continuous basis as it is manufactured. The full production volume of butter goes through the measurement cell. The near infrared beam passing through the butter measures a significant portion of the butter volume. This provides an excellent averaging of the parameters measured. The cell is easily cleaned and maintained in sanitary condition for the purpose of handling product for human consumption.

EXAMPLE III

Analysis of ground meat flowing through a pipe by Near infrared spectroscopy

Similar to example II above, example III shows the analysis of ground meat as it flows through a monolithic conveying conduit. The conduit is made from material that is transparent to infrared light such that quality parameters of meat can be determined continuously as the meat flows past a beam of infrared light that has distinct wavelengths or is modulated at unique frequencies according to each distinct wavelength (FT-NIR).

Fig.9 Distribution of light intensity verses wavelength through Monolithic conveying conduit made from PTFE filled with a variety of meat products including lean and fat Pork and lean and fat beef. The spectral region covers the near infrared from 7000 to 16000 cm-1 (1428nm to 625nm)

Fig. 10 shows a photograph of an installation of monolithic conveying conduit machined from a block of PTFE infrared transparent material for the analysis of flowing ground meat. The cable attached to the middle of the cell is a fiber optic cable, which transports infrared light that has been modulated with multiple unique frequencies corresponding to the wavelengths of the light as produced by an FT-NIR spectrometer. The light is measured by an infrared detector on the opposite side of the cell and decoded by means of a Fourier Transformation performed in a computer in order to produce a spectrum as shown in figure 1-ex3. The infrared spectra contain variations that vary linearly with the concentrations of different quality parameters such as % fat, % protein and % moisture. The concentrations are determined by a Partial Least Squares multivariate analysis.

While the invention has been described with particular reference to the illustrated embodiment, it will be understood that numerous

modifications thereto will appear to those skilled in the art. Accordingly, the above description and accompanying drawings should be taken as illustrative of the invention and not in a limiting sense.

WHAT IS CLAIMED IS:

1. A flow-through cell for optical analysis of a composition comprising:
   - at least one monolithic conveying conduit for transporting said composition;
   - at least one input light source for illuminating said composition through a section of the conveying conduit; and
   - at least one light receiving sensor, said light receiving sensor collecting light emerging through the illuminated composition and capable of transmitting received light transmitted through said composition to an analyzer operatively connected to produce an output indicative of the quantitative and/or qualitative level of light transmitted through said composition.

2. The flow-through cell of claim 1, wherein input light source is perpendicular to the conveying conduit.

3. The flow-through cell of claim 1, wherein said monolithic conveying conduit is made of a material selected from the group consisting of polytetrafluoroethylene, PTFE, fluorinated hydrocarbons, quartz, silica, sapphire, aluminosodic borosilicate glass, and transparent matter allowing passage of visible light, infrared and ultra violet radiations.

4. The flow-through cell as claimed in claim 1, wherein said light is infrared light.

5. The flow-through cell as claimed in claim 1, wherein said light is visible light.

6. The flow-through cell as claimed in claim 3, wherein said infrared is composed of wavelength in the range of 600 nm to 25 000 nm.

7. The flow-through cell as claimed in claim 1, wherein said composition is selected from the group consisting of liquid, gas, air, fluid, paste, foam, emulsion, gel, and powder.

8. The flow-through cell as claimed in claim 1, further comprising means for holding said conveying conduit in a desired position relatively to a light pathway traversing said conveying conduit.

9. The flow-through cell as claimed in claim 8, wherein said means for holding said conveying conduit can be variably positioned to adjustably tighten said conveying conduit.

10. The flow-through cell as claimed in claim 1, further comprising a mean for modulating the temperature of said composition before and/or during passage through a measuring section.

11. The flow-through cell as claimed in claim 1, wherein said monolithic conveying conduit allows for diffuse transmission of radiation of said input light source, so that only a fraction of transmitted light is intercepted by the light receiving sensor.

12. A method for measuring quantity or concentration of at least one component or physical property of a composition comprising:

c) capturing light emerging through an illuminated composition passing through a monolithic conveying conduit: and
d) comparing said captured light of step a) with said light before its passage through said composition to obtain a differential measure of light indicative of the quantity or concentration of a component or physical property of said composition.

13. The method of claim 12, where said monolithic conveying conduit is made of a material selected from the group consisting of polytetrafluoroethylene, PTFE, fluorinated hydrocarbons, quartz, silica, sapphire, aluminosodic borosilicate glass, and transparent matter allowing passage of visible light, infrared and ultra violet radiations.

14. The method of claim 12, wherein said light is infrared light.

15. The method of claim 12, wherein said light is visible light.

16. The method of claim 14, wherein said infrared is composed of wavelength in the range of 600 nm to 25 000 nm.

17. The method of claim 12, wherein said composition is selected from the group consisting of liquid, gas, air, fluid, paste, foam, emulsion, gel, and powder.

18. The method of claim 12, wherein said physical property is at least one of viscosity, firmness, consistency, or fluidity.

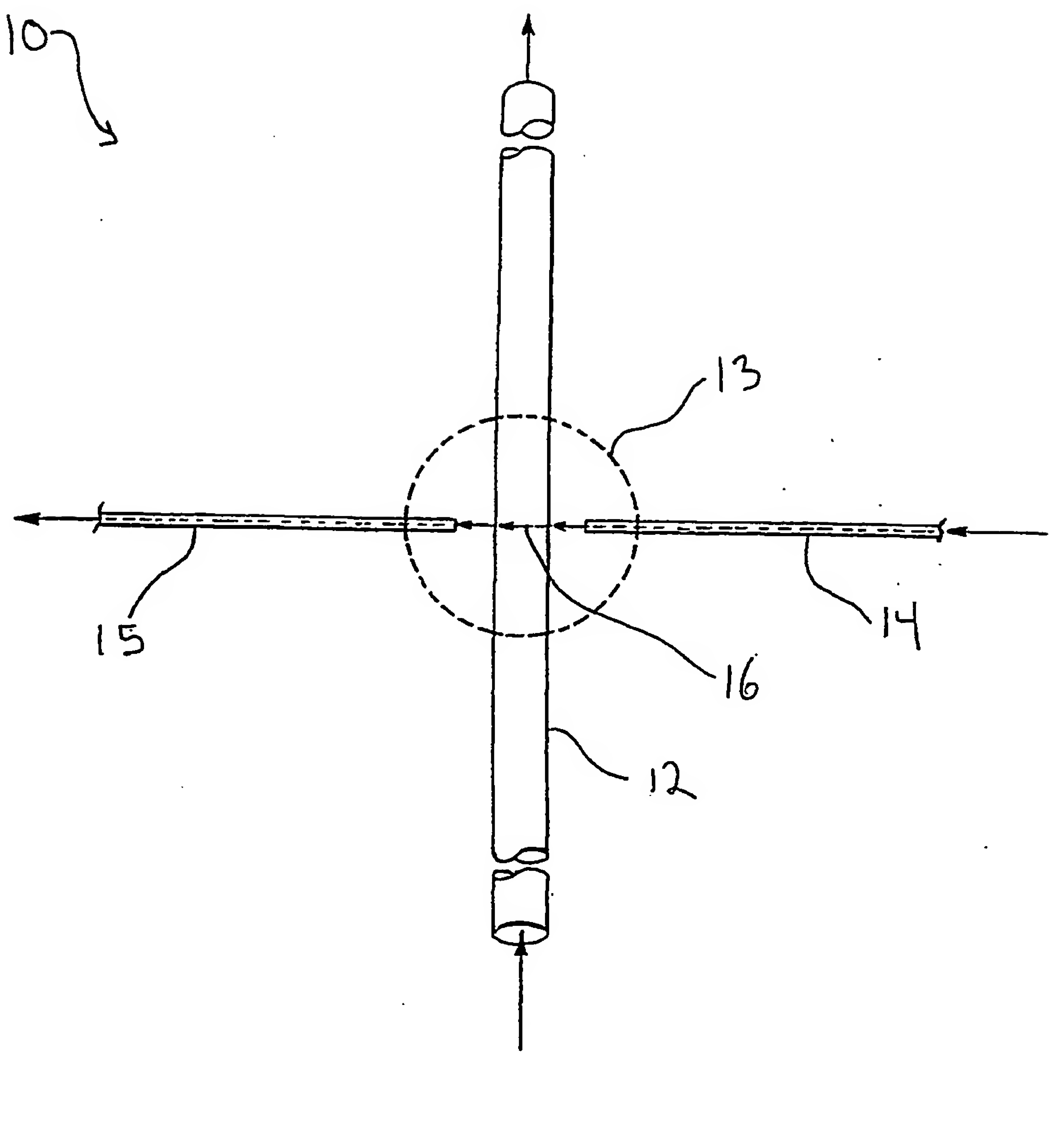

Fig. 1

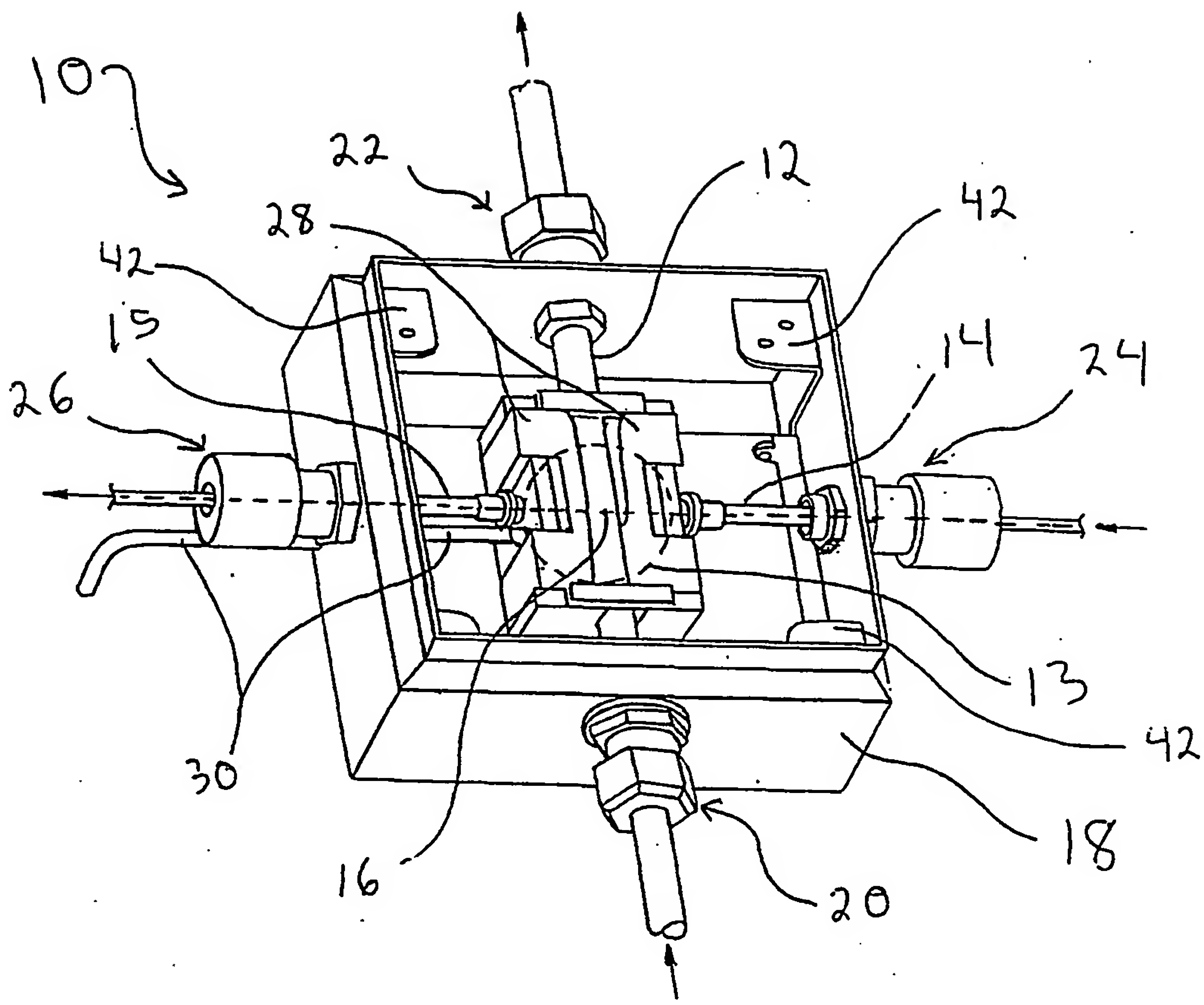

Fig. 2

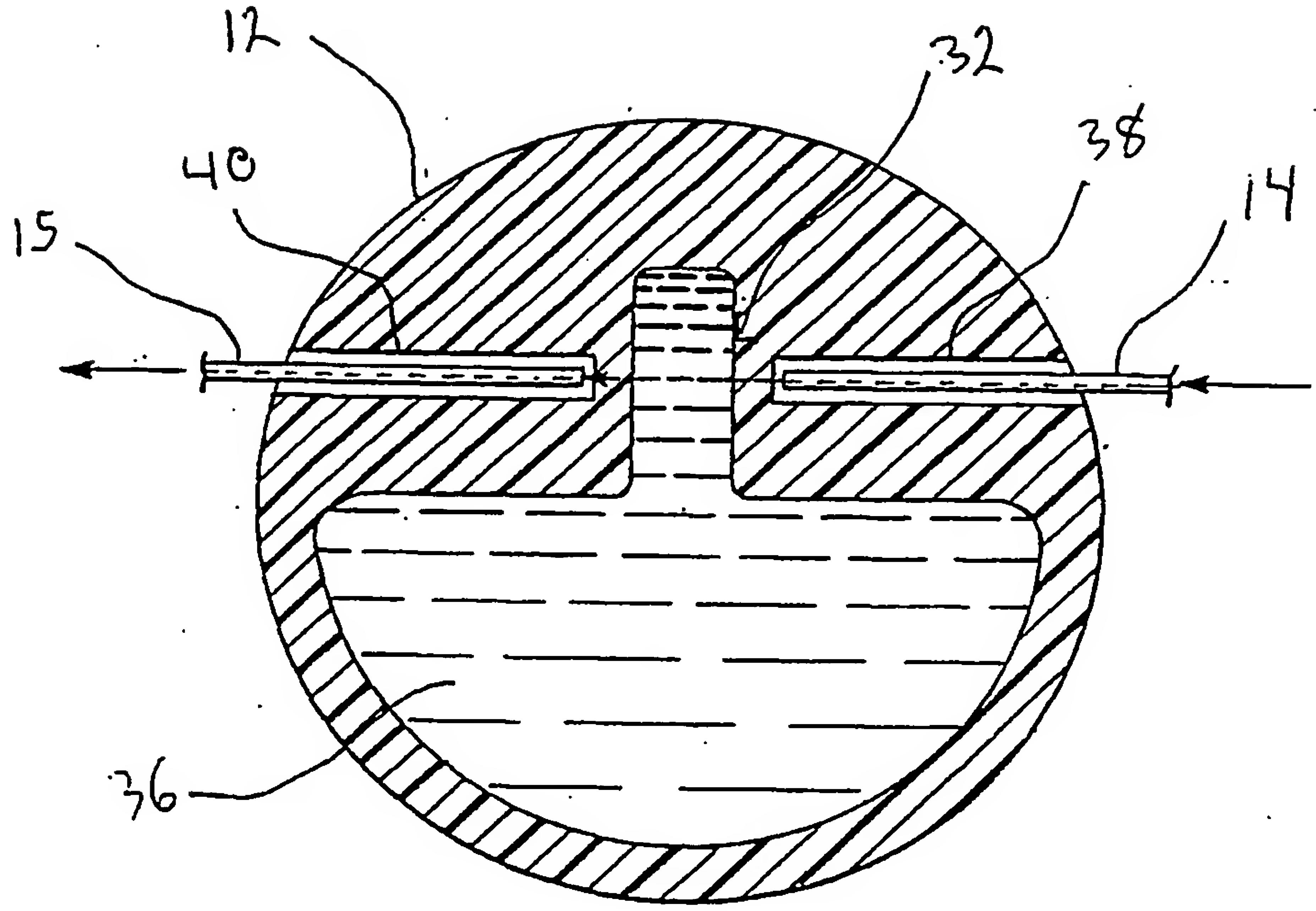

Fig. 3

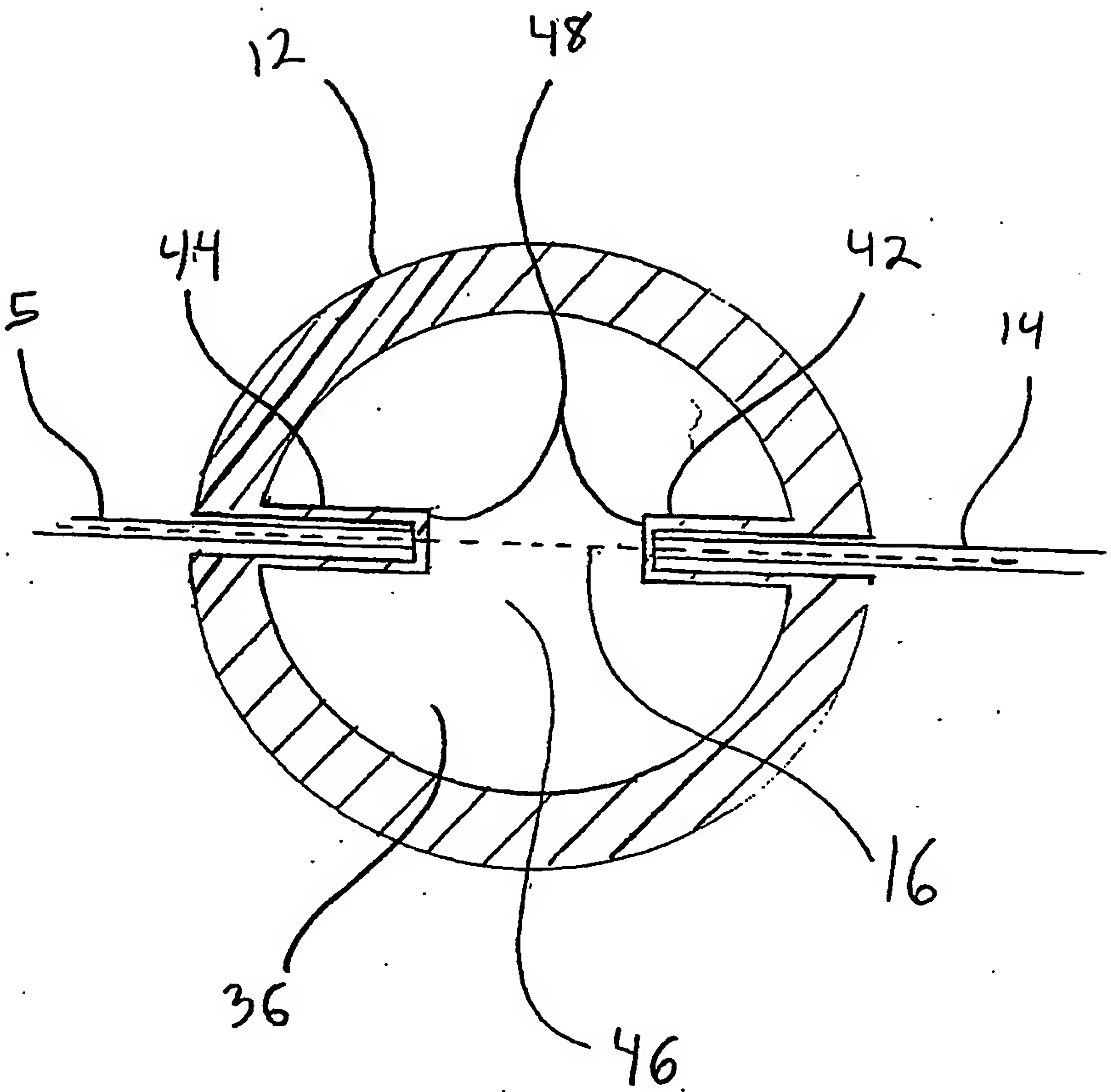

Fig. 4

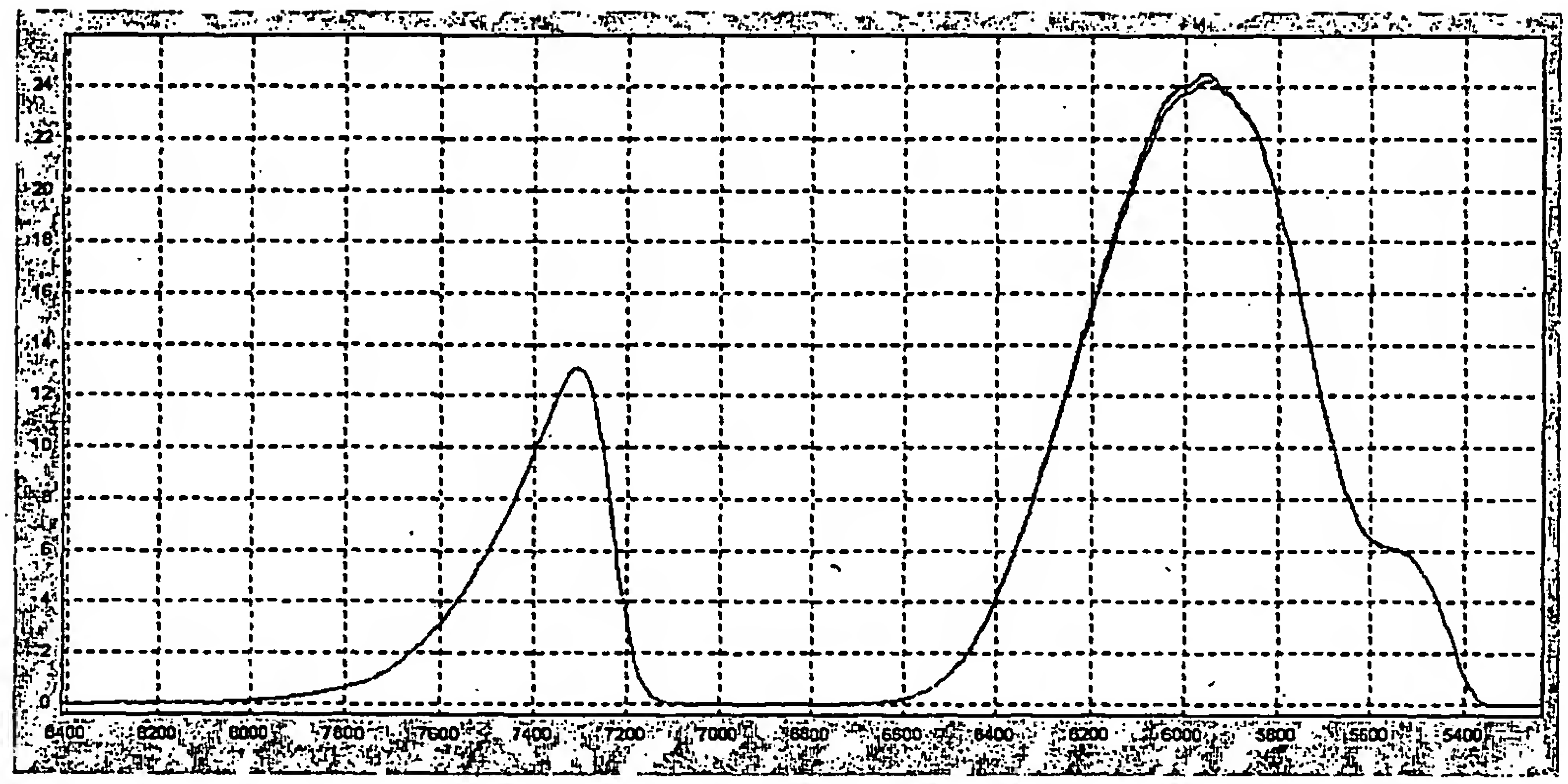
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Fig. 5

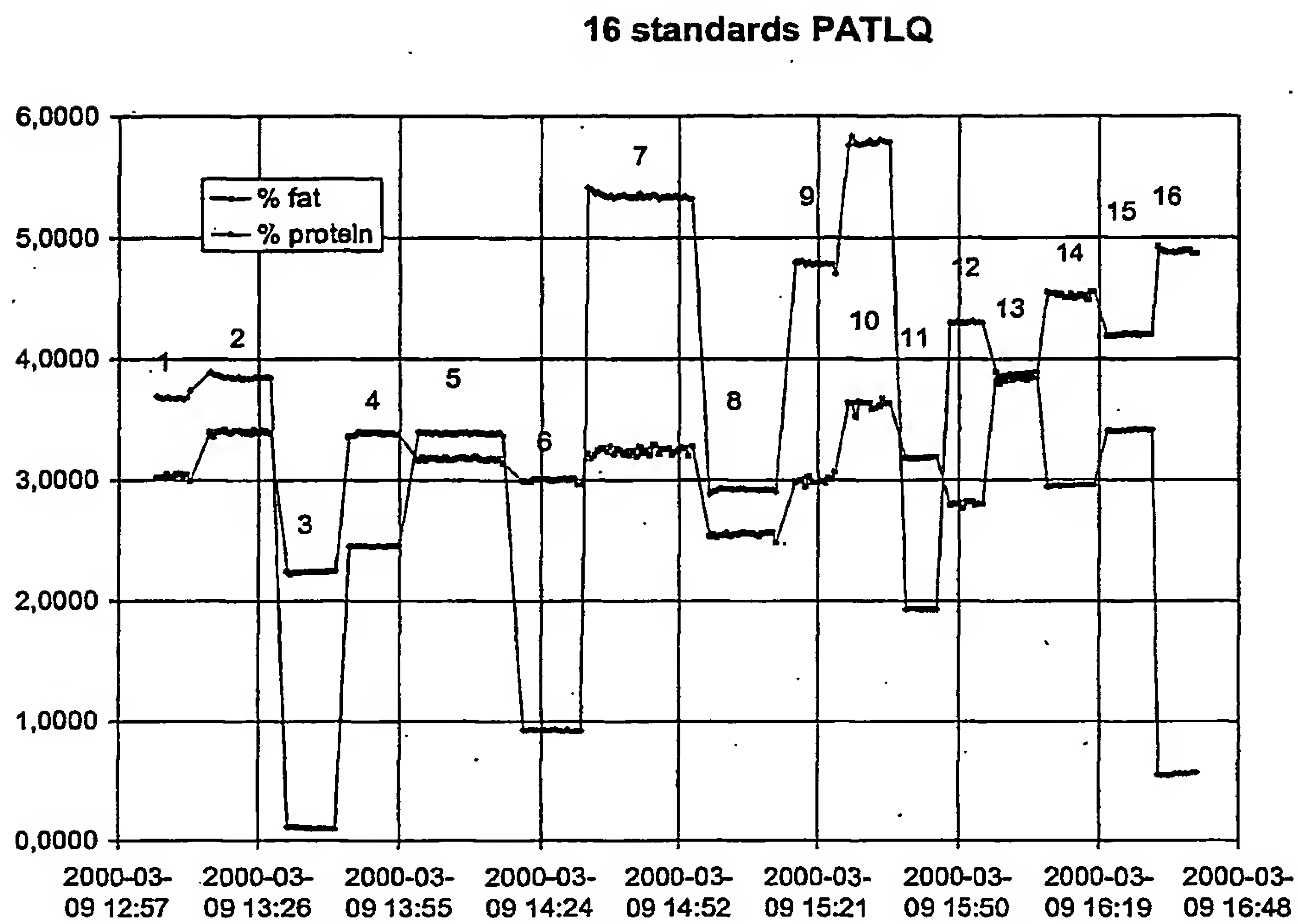
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Fig. 6

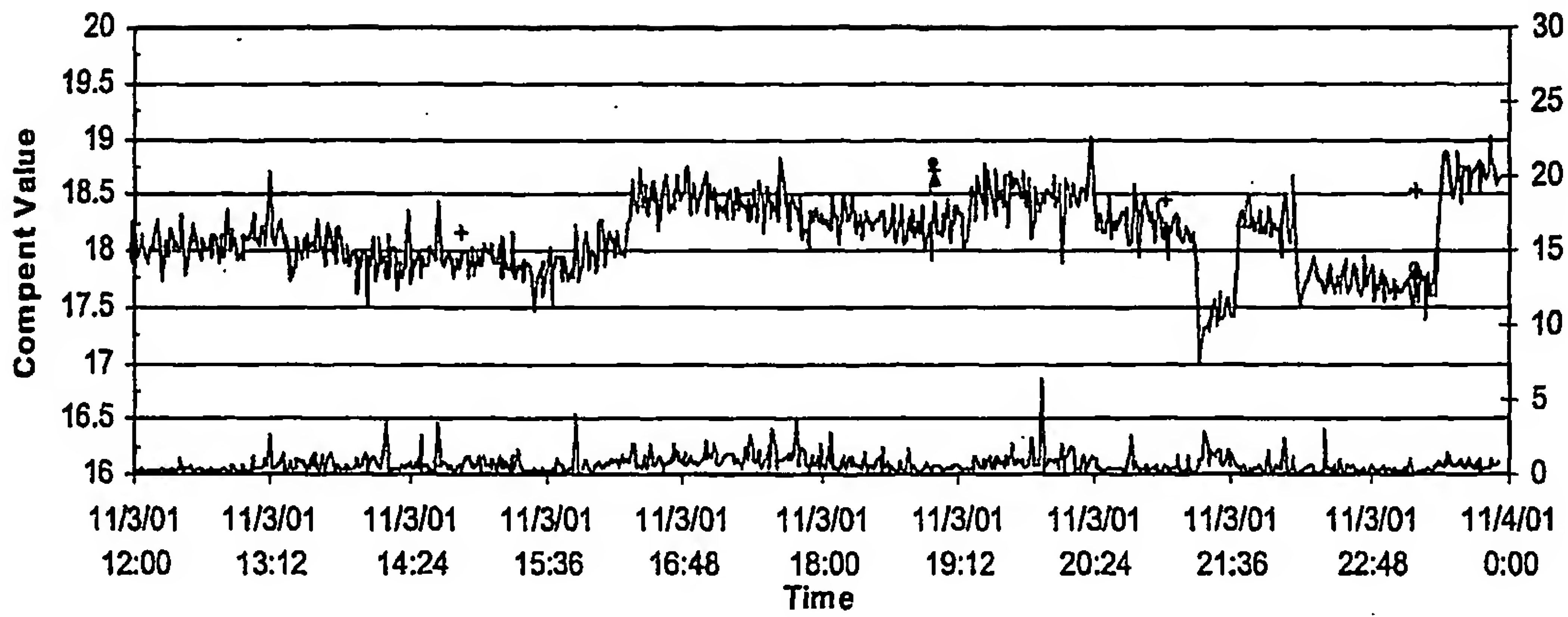

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Fig. 7

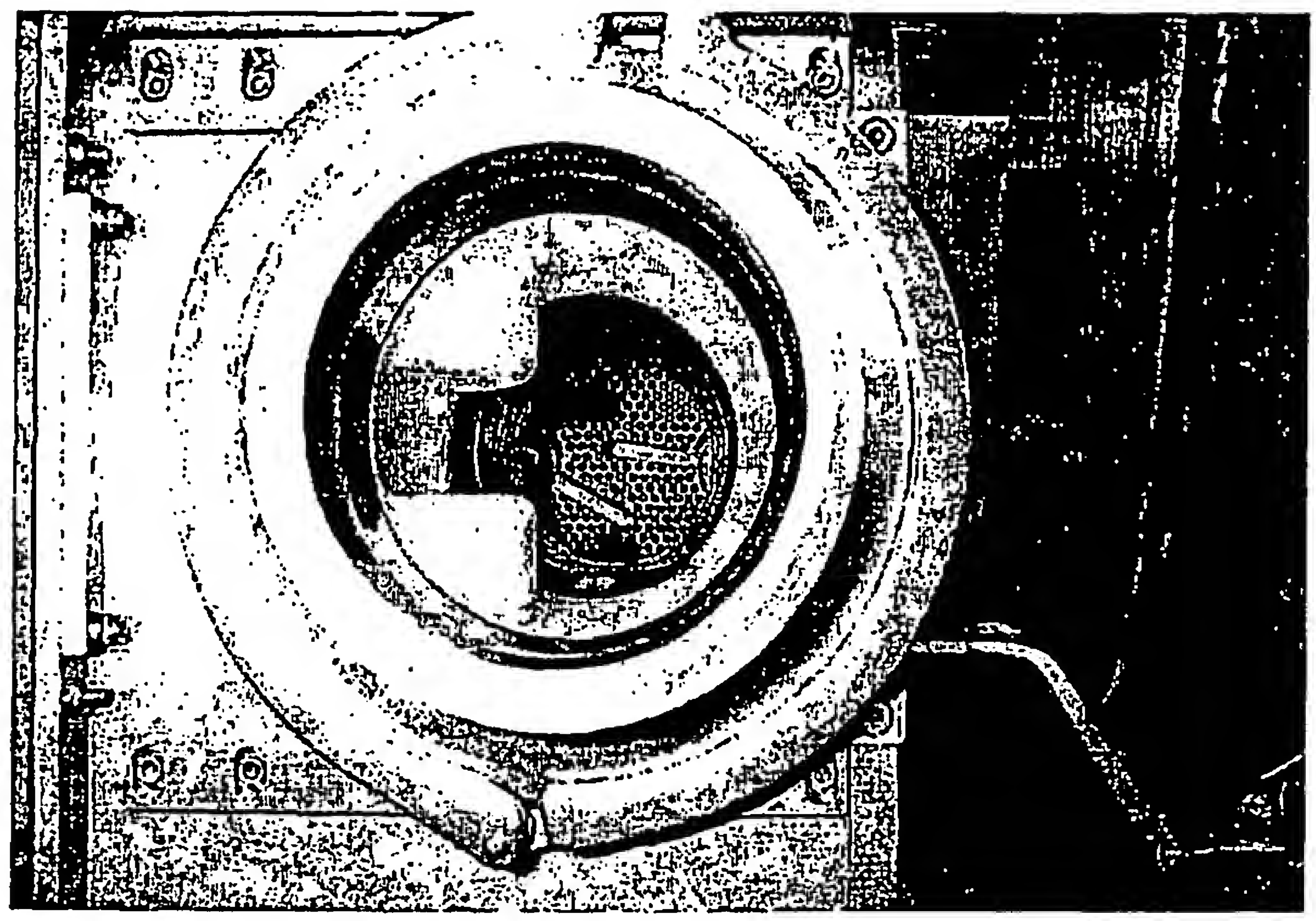

Fig. 8

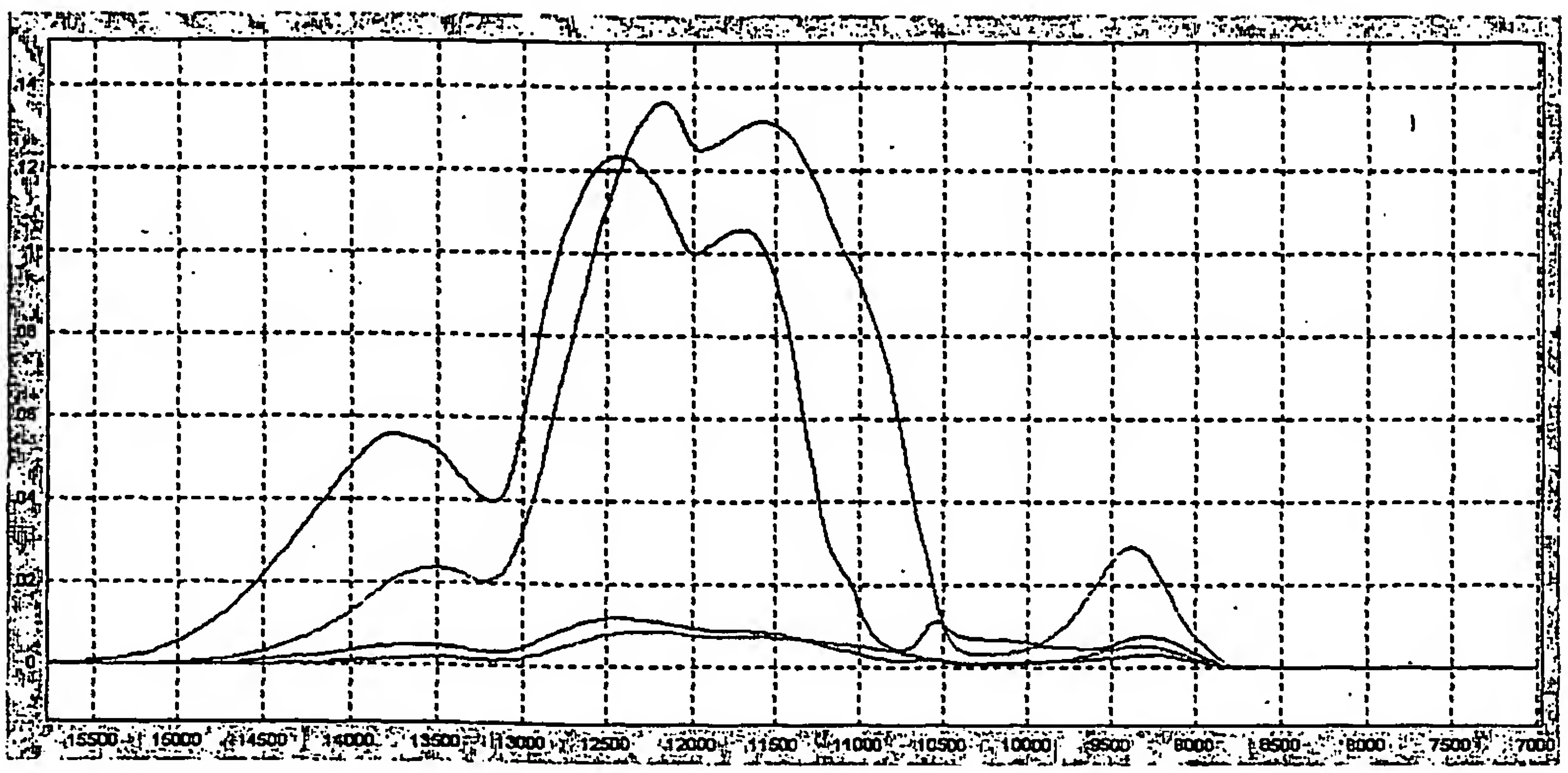
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Fig. 9

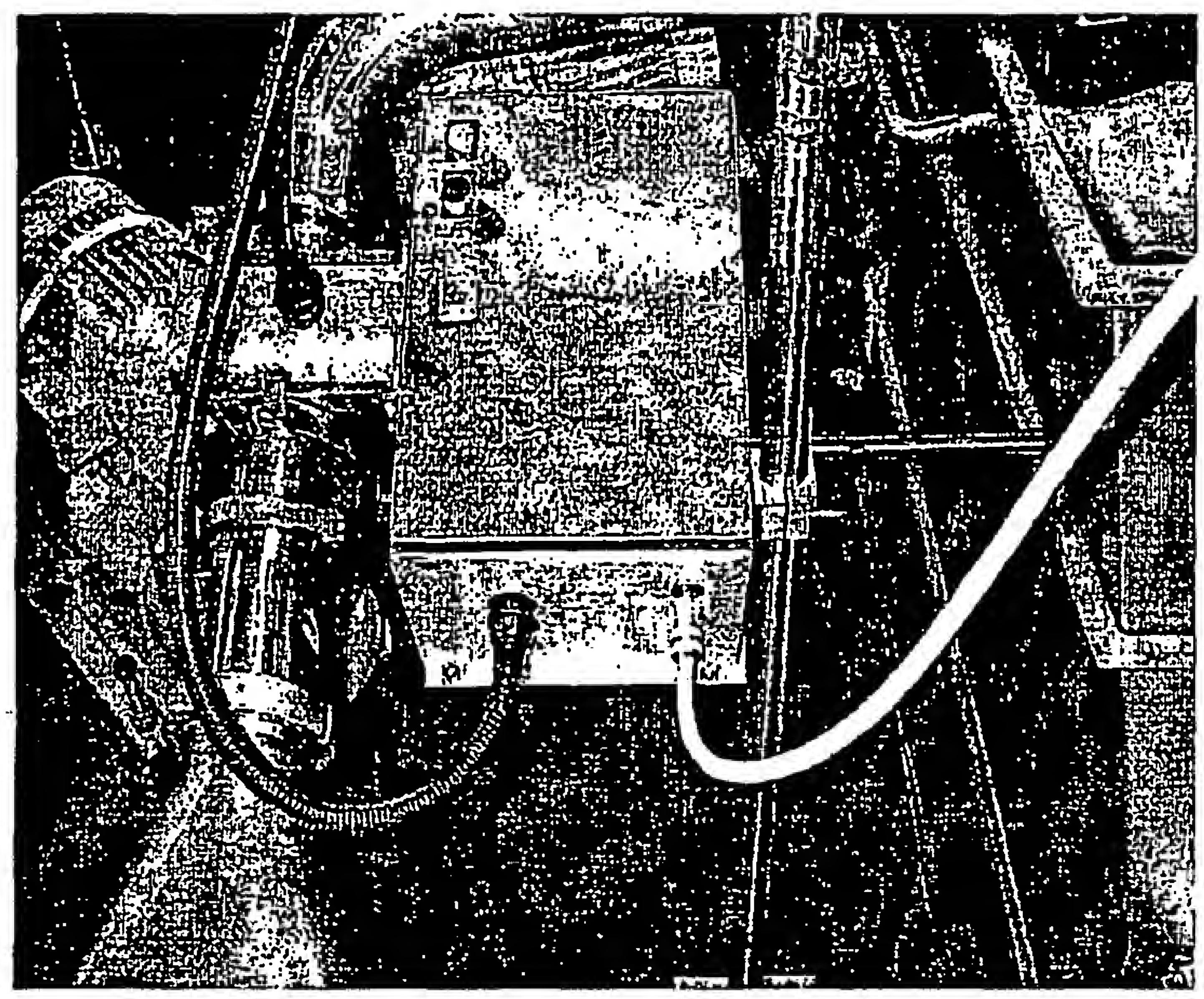

Fig. 10